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# Synthesis and properties of electroless Ni–P–Nanometer Diamond composite coatings

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#### Abstract

Nanometer Diamond (ND) was synthesized by using the detonation method. It was a kind of materials with properties of diamond and nanometer particle. The Ni-P-ND composite coating was prepared from a suspension of diamond nanoparticles in electroless bath. The tribological properties, hardness and corrosion behaviours of the composite coating on medium carbon steels were investigated. The Ni-P-ND composite coating exhibits not only high wear resistance but also low friction coefficient compared with the Ni-P composite coating. The highest microhardness of the Ni-P-ND composite coating was obtained by heat treated at 673 K. The corrosion resistance of Ni-P-ND composite coating is superior to that of Ni-P coating. The mechanisms of improvement of the tribological and electrochemical properties of the electroless composite coating are also discussed.

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#### 1. Introduction

Electroless Ni-P composite coatings have been widely used in many application fields for their unique combination of properties such as wear resistance, corrosion resistance, non-magnetism and uniformity of coating thickness. Choosing suitable particulate materials can easily produce functional composite coatings with highly specific characteristics. These solid particles such as silicon carbides, ceramics, fluoropolymers and aluminum oxide can be usually chosen to improve wear and corrosion behaviours [1-4].

Among the particulate materials used for reinforcement, since diamond has high hardness, good oxidation resistance and good chemical stability, diamond is frequently studied and applied. Reddy et al. [5] estimated the wear resistance of electroless Ni-P-C composite coatings before and after heat treatment and found that the wear resistance appears to be influenced by the size

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of the diamond particles. The Ni-P-C composite coatings formed by finer diamond particles are more wear resistant.

Nanometer Diamond (ND) was synthesized by the explosive detonation method and its structure and properties have been studied in the literature in recent years [6,7]. Its particles with an average grain dimension of 4-8 nm and specific surface area of 250-300 m<sup>2</sup>/g. Considering the spherical morphology and the particles size of the diamond, it was proposed that the electroless composite coating containing ND will show better properties than that of the traditional electroless diamond composite coatings. In this paper, the preparation and properties of Ni-P-ND electroless composite coating are described. The results demonstrate that the Ni-P-ND composite coatings exhibit better tribological performances than the Ni-P coatings. Microhardness of the Ni-P-ND composite coatings before and after heat treatment are also reported. The corrosion behaviour of amorphous deposits is also evaluated by polarization curves in NaCl 0.1 M solution at room temperature. It is noted that the amorphous structure Ni-P-ND composite coating provides higher corrosion resistance compared with the amorphous structure Ni-P coating.

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# 2. Experimental

Medium carbon steel samples of 24 mm diameter and 8 mm thickness were metallographically prepared using 1000-grade SiC paper and ultrasonically cleaned in acetone The treatment process used prior to deposition consisted of three different steps: (1) cleaning with NaOH–Na<sub>2</sub>CO<sub>3</sub> solution at 353 K; (2) cleaning with acetone in an ultrasonic bath for 10 min; (3) electrolytic activation in dilute H<sub>2</sub>SO<sub>4</sub>. Between every step the samples were rinsed in distilled water. Prior to plating, the surface roughness of the steel substrate was approximately 0.4 μm mm (*Ra*).

The electroless plating were carried out in a cell placed in a thermostatically controlled bath. The bath compositions and conditions for the electroless coatings were: nickel sulfate 28–30 g/l, sodium hypophosphite 20–25 g/l, sodium acetate 10–15 g/l, propionic acid–lactic acid 10 ml/l, surfactant 200–400 mg/l, pH=4.5. The plating bath was maintained at a constant temperature of 361 K. Ni–P coating was firstly deposited for 0.5 h and then ND suspended in the plating bath was deposited for 2.5 h. For comparison, the Ni–P coating was deposited on the samples according to a similar procedure. The thickness of the deposit was in the range of 28–30 μm in all cases.

ND used in this test were immerged in a concentrate nitric acid for 24 h, then washed with distilled water till pH=7 and dried at 393 K in vacuum, ND with 98% purity were obtained.

Because ND particles were very easy to agglomerate to form large particles for their high surface energy, it is noted that the dispersion clusters of nanometer diamond particles is very important for the preparation of nanostructure composite coatings. During the co-deposition ND particles into electroless Ni-P coating, the role of the surfactant dispersant is not negligible. Surfactant dispersant can not only improve the stability of a suspension by increasing the wettability and the surface charge of suspended particles which prevent them from agglomerating but also enhance the electrostatic adsorption of suspended particles on a substrate. At the same time, in order to improve the dispersion of ND in electroless bath, the purified ND were milled for 8 h with a Planetary ball mill machine in a few surfactant liquids at a rotating speed of 1000 rev/min. Then the ND powder of a calculated quantity was mixed thoroughly with quantity mixture surfactant and 10 ml of the plating solution, ultrasonically dissolved 30 min and then transferred to the plating bath. An enhanced agitation system was employed during the deposition process for all the samples under study. Argon was injected through the bottom of the cell. This agitation not only permitted production of a uniform and defect-free surface but also helped to maintain the ND particles in suspension. Both bath agitation and blending surfactant allow the break up of agglomerates particles, which is necessary to obtain homogeneous composite coatings containing a fine dispersion of the nanoparticles.

In this work, Microstructural analysis of the electroless composite coatings were identified by Rigaku D/MAX-2400 diffractometer with CuKα radiation X-ray. The surface morphology of the electroless composite coatings was evaluated using Atomic Force Microscope (AFM). Microhardness of the coatings was determined with VDMH-5 Version 2.01 hardness indenter using a load of 100 g and a time of 5 s. Five readings were taken on each deposit and then the values were averaged. The SRV reciprocating ballon disc wear testing machine (Germany) was carried out to examine the tribological properties of the composite coatings in dry non-lubricated conditions. The upper ball specimen was made from GCr15 (hardness: 62HRC) with a diameter of 10 mm. The wear volume was calculated based on the profiles of the wear tracks measured by profilometer. The friction coefficients of Ni-P coating and Ni-P-ND composite coatings were simultaneously recorded. The average values of the friction coefficients were obtained under stable conditions in all tests. The experiments conditions were: vertical load 20 N, frequency 10 Hz, amplitude 1 mm, time 20 min. The lost volume of the specimen after wear test was used as a measurement to evaluate the wear resistance of coatings. Corrosion behaviour was estimated from potential and polarization measurements on the electroless films. The films were prepared directly on plates of surface area 8 cm<sup>2</sup> without any special preparation. The electrolyte used was 0.1 M NaCl solution. The electrochemical properties of the composite coatings were investigated using CHI-614 electrochemical workstation. A saturated calomel electrode (SCE) was used as a reference electrode.

## 3. Results and discussion

A transmission electron microscopy (TEM) image of purified nanometer diamonds synthesized by the detonation method is shown in Fig. 1. From the figure, it can be seen that the morphology of the diamond powder is close to sphere and the diameter in the range of 3–8 nm. Obviously,

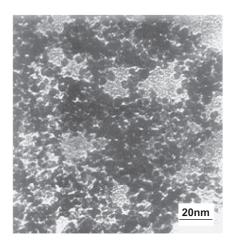


Fig. 1. TEM image of ND.

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